

## Standard Operating Procedure for Zeiss EVO-50 ESEM (10/18/10)

1. Turn on computer monitors (rightmost button on each).
2. Enter username and password to logon to the NEST lab LAN.
3. Note if there are any conditions in the login screen that are highlighted in red – these are conditions that should be brought to the attention of NEST lab personnel.
4. Enter username and password (wittbetn, nest123) to logon to the SmartSEM software.
5. Ctrl G will display SEM control information; drag this window to right screen; this window contains 6 tabs – Gun, Detectors, Aperture, Scanning, Stage, and Vacuum.
6. To insert sample, click Vent on right side of lower toolbar in SmartSEM window.
7. Click Yes when asked “Do you want to Vent.” After ~30 sec a valve will open automatically and air will be admitted to the sample chamber and electron beam column.
8. Remove old sample stub by loosening setscrew on sample stage; insert new sample stub and tighten setscrew. Note that the sample stage moves to the lowest z-axis position during venting. **If sample is large, be certain that there is adequate clearance between sample, gun aperture, and EDAX detector before closing sample door.**
9. Close sample door. Check Vacuum tab. If you are analyzing a non-conductive sample, click on Go to VP. This will allow some air into the sample chamber while pumping the electron gun to high pressure. The pressure in the sample chamber is adjustable. Usually a pressure of 10 Pa is sufficient to neutralize charging on non-conductive samples. Click Pump to evacuate the instrument.
10. Vacuum conditions can be monitored with the Vacuum tab in the SEM control window. When the vacuum in the electron gun is  $< 1 \times 10^{-4}$  Torr, click Beam On in the lower right corner of the main SEM image window. The Gun parameters can be monitored with the Gun tab of the SEM control window on the right screen. Typical operating conditions are 20 kV beam energy, 2.68 A filament current, and a spot size of ~350 – 450. The filament current is optimized when a new filament is installed. **Do not increase filament current as this could greatly reduce filament life.**
11. All adjustments to the SEM image can be made with the mouse. Moving the mouse to the left while holding down the left or middle button will decrease the parameter corresponding to that button. Moving to the right will increase that parameter.
12. **If you are having difficulty getting an SEM image, in the Aperture tab, click on Emission and make sure the electron beam is centered using the Gun Shift and Tilt buttons. Click on Normal after completing electron beam alignment.**

13. Click on the rightmost icon on the toolbar above the SEM image in order to control the Mag / Focus. The magnification will be controlled with the left mouse button. The magnification can be read on the lower toolbar highlighted in green. Left-clicking on this green box will bring up a dialog box so that a particular magnification can be entered. The focus is controlled with the middle mouse button and is read as working distance highlighted in yellow on the lower toolbar.
14. The Brightness/Contrast is adjusted by clicking on the second button from the right on the upper toolbar. The contrast will be controlled by holding down the middle button on the mouse and moving it to the left or right while the arrow is over the SEM image. The brightness is controlled with the left mouse button. The % brightness and % contrast can be read on the lower toolbar.
15. The stigmation is adjusted by clicking on the third button from the right on the upper toolbar. Holding down the left mouse button and moving the mouse from side to side adjusts the x-stigmation. Holding down the middle mouse button allows adjustment of the y-stigmation.
16. The distance between the sample and the electron gun aperture (working distance) should be set according to the information that is required by adjusting the z-position. **Always enable the chamberscope (icon near middle of top toolbar) when adjusting the z-position. Running the sample into the bottom of the electron beam column will cause expensive damage to the instrument!** The working distance should be ~5-10 mm for analysis requiring high spatial resolution. For EDAX analysis, the optimal working distance is 15 mm.
17. The electron beam energy and probe current (spot size) should be adjusted according to the sample and the results required. These parameters can be viewed and adjusted in the Gun tab. A spot size ~250 is good for high spatial resolution, but may give noisy images unless slow scan times are selected. A spot size of 350 – 500 will give good x-ray intensity for EDAX analysis and is usually adequate for images up to ~2000x. **Do not click AutoSat.**
18. Scan rate and frame averaging can be adjusted in the Scanning tab. A scan rate of 3 or 4 gives a relatively fast rate that is good for locating an area of interest and doing initial focus adjustments. A scan rate of 7 or more will give a better quality image.
19. Information about image conditions, including a micron marker, can be added to the bottom of an image and will be stored along with the image file. On the task bar, select View, Datazone and check Unhide.
20. A photo of a particular area can be obtained by clicking on Photo on the top toolbar. This image can be saved in a variety of formats by right-clicking on the image. Be sure to save images to the y-drive so that they can be retrieved from the computer in the large outer area after your analysis session is completed.

21. The Zeiss SEM can be fitted with apertures to allow operation in high vacuum mode or in several variable pressure modes. The typical configuration is with the 750  $\mu\text{m}$  final aperture one VP aperture installed. This allows operation in either the high vacuum mode or in the VP mode at pressures up to 400 Pa.
22. If the 20 or 30  $\mu\text{m}$  final aperture is used, it may be necessary to adjust it's position. This can be done by clicking Focus Wobble in the Aperture tab, and adjusting the x and y micrometers on the column to stop movement of the SEM image. This should be done carefully since it can be difficult to find the image if these are misadjusted.